
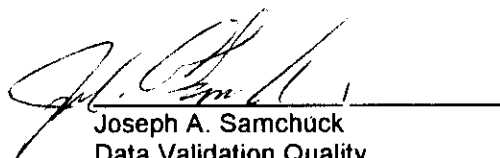


Data Limitations and Validation Report  
for Environmental Groundwater Samples  
Collected from the Argonne National Laboratory - West  
Scoville, ID  
Case No. 93081806  
SDG. No. 93081806  
Target Analyte List (TAL) Metals plus Tin  
Three Aqueous Samples

Validated by:

  
Ricky C. DePaul  
Data Validation  
Reviewer

Approved by:

  
Joseph A. Samchuck  
Data Validation Quality  
Assurance Officer

A. TITLE:

INORGANIC DATA LIMITATIONS and VALIDATION REPORT

Project Site: Waters from Argonne National Laboratory - West  
Sample Type: Aqueous samples  
Analysis Type: TAL Metals plus Tin  
Case No.: 93081806  
SDG. No.: 93081806

B. INTRODUCTION:

A complete review, following the procedures outlined in SMO-SOP-12.1.5<sup>1</sup>, was performed on the data package, labeled Case No. 93081806, SDG# 93081806, submitted by Biospherics Incorporated. Based upon the information available for review, it appears as though, the laboratory analyzed the aforementioned water sample from the Argonne National Laboratory - West according to SW846 analytical protocols. The deliverable format does not comply with data package requirements pursuant with Level A validation protocol. The review could not include Level A data validation confirmation.

C. CONTRACT AND TECHNICAL REVIEW:

Site: Water from Argonne National Laboratory - West  
Type: TAL Metals plus Tin  
Case No.: 93081806  
SDG No.: 93081806  
Laboratory: Biospherics Incorporated

Sample Identification:

<u>FIELD ID</u>	<u>LAB ID</u>
ANL-158-93	93081806-1
ANL-168-93	93081806-2
ANL-177-93	93081806-3

CTR COMMENTS:

1. Samples were collected on 7/28/93. Inductively Coupled Plasma (ICP) and Graphite Furnace Atomic Absorption (GFAA) analytes were analyzed approximately two months after sample collection. Hence, no validation actions were warranted for these analytes. Mercury analyses appear to have been conducted approximately 28 days after sample collection. The laboratory also indicates reported results for mercury as noted by the 09/17/93 analyses as indicated by the use of the "X" laboratory flag. It appears as though the laboratory incorrectly used the "X" flag as noted on the Form XIV. The validator amended the appropriate form.

The results for mercury were not correctly reported as noted on the Form Is. It appears as though the laboratory actually reported the absorbance values for this analyte in these samples. The validator amended the appropriate Form Is as per the actual raw data results (i.e., < 0.5 ug/L for each sample). The laboratory reports to a detection limit of 0.5 ug/L, which is not the detection limit specified in the associated Statement of Work (SOW) for these samples. To summarize, the laboratory incorrectly reported the mercury results to a detection limit above the level of detection which was specified for this analyte as noted in the SOW. These types of problems are indicative of the anomalies being noted during validation review for these data packages. These types of mistakes are unacceptable and render the data highly suspect.

2. Portions of the raw data were illegible and provided no useful information. It was not possible for the

validator to verify reported sample results as per Level A validation review criteria. This is noted here for completeness.

3. A Continuing Calibration Verification (CCV) Percent Recovery (%R) for tin was excessively high as verified by the validator against the raw data. The laboratory chose not to report a %R for this analyte as noted on the Form 2A. It appears as though only nondetects were reported for this analyte in these samples.
4. The sample data were not evaluated for blank contamination as it is the professional opinion of the data reviewer that such an evaluation would not lend added value to the data. Dilution factors were not chronicled on the Form XIVs as necessary for proper evaluation of this parameter.

Additionally, the absence of reporting limits and the practice of reporting results below SOW reporting limits compromised the blank evaluation process. The data reviewer could not therefore evaluate blank contamination in accordance with SMO-SOP-12.1.5<sup>1</sup>.

The laboratory incorrectly reported to the instrument response level as noted in the raw data and included in the support documentation. This is grossly incorrect and substantially biases the data set in a statistical sense. The laboratory reported negative values as noted on the Form I. The laboratory must report instrument response readings (which are sufficiently low as to yield results below the established instrument detection limit) to the IDL and not report values which are noted in the "raw" instrument print-out. The validator cites the reporting limit of 2.5 U ug/L for arsenic as noted in sample ANL-158-93. The final concentration for this analyte was 5.0 U ug/L.

5. The laboratory incorrectly reported to the instrument response level as noted in the raw data and included in the support documentation regarding Graphite Furnace Atomic Absorption (GFAA) analyses as noted above. This is grossly incorrect and substantially biases the data set in a statistical sense. The laboratory must report instrument response readings (which are sufficiently low as to yield results below the established instrument detection limit) to the IDL and not report values which are noted in the "raw" instrument print-out. The validator can also cite the reporting limit of 2.1 ug/L for selenium as noted in sample ANL-158-93. The final concentration for this analyte as noted in the raw data was < 5.0 ug/L.
6. Similarly, the laboratory incorrectly reported mercury results as noted for the samples included in this SDG. The validator has included sufficient raw data support documentation to verify this claim as noted for mercury in samples ANL-158-93, ANL-168-93, and ANL-177-93. These anomalies severely compromise the data and render it nonusable. Specific incorrectly reported absorbances for these analytes were 0.002, 0.006, and 0.003, respectively.
7. The laboratory did not adequately complete the ICP Interference Check Sample (ICS) Form IV. One of the purposes of analyzing the ICP ICS solution is to determine the potential impact of the four interfering analytes on potentially impacted analytes. This was not done. The interfering analytes aluminum, calcium, iron, and magnesium were noted reported for this solution. No problems were noted with the reported ICSAB solution recoveries. However, recoveries were not reported for chromium and zinc as noted on the Form IV. These ICSAB found values for these analyses appear to yield acceptable recoveries. It is noted that interfering analytes were present in the environmental samples at sufficiently low levels as to not introduce interference affects.
8. The Matrix Spike (MS) Form 5A and laboratory duplicate Form 6 were erroneously reported with numerous reporting inconsistencies rendering them useless without major revisional incorporations. The sample data were not further evaluated for these parameters.
9. The aqueous Laboratory Control Sample (LCS) recoveries for numerous analytes were not reported in some instances. Furthermore, the aqueous LCS found values for calcium, magnesium, and sodium were not reported. Thus, this quality control parameter provides no useful information regarding data usability.

D. DATA LIMITATION OVERVIEW:

a. Summary of Qualified Data

Sample ANL-158-93 could not be fully evaluated given the limitations of the data package deliverable. Sample data qualifications were not made for the aforementioned quality control noncompliances (anomalies) as it is not possible to ascertain a cumulative affect of the type or severity of problems impacting sample data quality based upon the unacceptable format of the data package deliverable.

Sample ANL-168-93 could not be fully evaluated given the limitations of the data package deliverable. Sample data qualifications were not made for the aforementioned quality control noncompliances (anomalies) as it is not possible to ascertain a cumulative affect of the type or severity of problems impacting sample data quality based upon the unacceptable format of the data package deliverable.

Sample ANL-177-93 could not be fully evaluated given the limitations of the data package deliverable. Sample data qualifications were not made for the aforementioned quality control noncompliances (anomalies) as it is not possible to ascertain a cumulative affect of the type or severity of problems impacting sample data quality based upon the unacceptable format of the data package deliverable.

E. LABORATORY APPRAISAL:

The data package was presented in a format which could not be fully evaluated as per the validation review requirements as defined by Level A validation review criteria. Qualifications applied to the data serve to indicate problems which could effectively be identified based upon specific noncompliant quality control parameters. Various anomalies and inconsistencies prevented a logical and systematic evaluation process of identifying and qualifying analytical results with a given amount of certainty. The following notable items illustrate the systematic problems associated with this deliverable:

- inconsistent reporting of analytical results (i.e., results reported both above and below detection limits referenced in the SOW)
- negative results reported on Form Is
- absence of laboratory qualifications
- omissions of various analytes on various quality control summary forms

Furthermore, deficiencies noted with data presentation and reporting may not preclude additional, more severe problems with the data which could in affect render the data nonusable. It is not possible to make an accurate and complete assessment of the data. Overall data usability cannot be appraised for this data set as a result of problems noted with the deliverable.

F. REFERENCES:

1. Standard Operating Procedure For Inorganic Data Validation, "SMO-SOP-12.1.5", Environmental Restoration Program, EG&G, Inc., 1991.

**APPENDIX A**  
**RESULTS AS REPORTED BY THE LABORATORY**

U.S. EPA - CLP  
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INORGANIC ANALYSIS DATA SHEET

SAMPLE NO.

ANL-158-93  
L00061

Lab Name: BIOSPHERICS INCORPORATED

Contract: ARGONNE

Lab Code: 81806-1 Case No.: 93081806

SAS No.: \_\_\_\_\_

SDG No.: \_\_\_\_\_

Matrix (soil/water ): WATER

Lab Sample ID: 93081806-1

Level (low/med): MEDIUM

Date Received: 08/18/93

% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	7 U			P
7440-36-0	Antimony	60 U			P
7440-38-2	Arsenic	2.5 U	LS		F
7440-39-3	Barium	17.7 U			P
7440-41-7	Beryllium	-1.7 U			P
7440-43-9	Cadmium	8.9 U			P
7440-70-2	Calcium	39401			P
7440-47-3	Chromium	4.5 U			P
7440-48-4	Cobalt	0.3 U			P
7440-50-8	Copper	11.5 U			P
7439-89-6	Iron	9 U			P
7439-92-1	Lead	3.5 U			F
7439-95-4	Magnesium	13585			P
7439-96-5	Manganese	-8.8 U			P
7439-97-6	Mercury	0.002 U	0.5 U		CV
7440-02-0	Nickel	18.9 U			P
7440-09-7	Potassium	3010			P
7782-49-2	Selenium	2.1 U	LS		F
7440-22-4	Silver	-2.7 U			P
7440-23-5	Sodium	17764			P
7440-28-0	Thallium	-4.6 U			F
7440-62-2	Vanadium	4.3 U			P
7440-66-6	Zinc	31.4			P
	Cyanide				NR
7440-31-5	Tin	4.4 U			P

Color Before: \_\_\_\_\_

Clarity Before: \_\_\_\_\_

Texture: \_\_\_\_\_

Color After: \_\_\_\_\_

Clarity After: \_\_\_\_\_

Artifacts: \_\_\_\_\_

Comments:

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U.S. EPA - CLP  
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INORGANIC ANALYSIS DATA SHEET

SAMPLE NO.

ANL-168-93  
L00062

Lab Name: BIOSPHERICS INCORPORATED Contract: ARGONNE

Lab Code: 81806-2 Case No.: 93081806 SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Matrix (soil/water): WATER Lab Sample ID: 93081806-2

Level (low/med): MEDIUM Date Received: 08/18/93

% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	-38	U		P
7440-36-0	Antimony	60	U		P
7440-38-2	Arsenic	1.2	U	25	F
7440-39-3	Barium	16.1	U		P
7440-41-7	Beryllium	-1.7	U		P
7440-43-9	Cadmium	7	U		P
7440-70-2	Calcium	37457			P
7440-47-3	Chromium	10.4	U		P
7440-48-4	Cobalt	-1.5	U		P
7440-50-8	Copper	4.1	U		P
7439-89-6	Iron	42	U		P
7439-92-1	Lead	6.7	U		F
7439-95-4	Magnesium	12981			P
7439-96-5	Manganese	-4.9	U		P
7439-97-6	Mercury	0.006	U	254	CV
7440-02-0	Nickel	19.4	U		P
7440-09-7	Potassium	2960			P
7782-49-2	Selenium	2.6	U		F
7440-22-4	Silver	-0.7			P
7440-23-5	Sodium	17010			P
7440-28-0	Thallium	-5.4	U		F
7440-62-2	Vanadium	12.1	U		P
7440-66-6	Zinc	12.2	U		P
	Cyanide				NR
7440-31-5	Tin	7	U		P

Color Before: \_\_\_\_\_ Clarity Before: \_\_\_\_\_ Texture: \_\_\_\_\_

Color After: \_\_\_\_\_ Clarity After: \_\_\_\_\_ Artifacts: \_\_\_\_\_

Comments: \_\_\_\_\_

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U.S. EPA - CLP  
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INORGANIC ANALYSIS DATA SHEET

SAMPLE NO.

ANL-177-93  
L00063

Lab Name: BIOSPHERICS INCORPORATED Contract: ARGONNE

Lab Code: 81806-3 Case No.: 93081806 SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Matrix (soil/water ): WATER Lab Sample ID: 93081806-3

Level (low/med): MEDIUM Date Received: 08/18/93

% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	-11	U		P
7440-36-0	Antimony	60	U		P
7440-38-2	Arsenic	0.8	U	<5	F
7440-39-3	Barium	18.8	U		P
7440-41-7	Beryllium	-1.4	U		P
7440-43-9	Cadmium	4	U		P
7440-70-2	Calcium	38354			P
7440-47-3	Chromium	-0.9	U		P
7440-48-4	Cobalt	-7.3	U		P
7440-50-8	Copper	7.9	U		P
7439-89-6	Iron	-11	U		P
7439-92-1	Lead	0.2	U		F
7439-95-4	Magnesium	13337			P
7439-96-5	Manganese	56.1	U		P
7439-97-6	Mercury	0.003	U		CV
7440-02-0	Nickel	-2.2	U		P
7440-09-7	Potassium	3030			P
7782-49-2	Selenium	3.5	U	<5	F
7440-22-4	Silver	-3	U		P
7440-23-5	Sodium	17716			P
7440-28-0	Thallium	-1.7	U		F
7440-62-2	Vanadium	9.5	U		P
7440-66-6	Zinc	7.8	U		P
	Cyanide				NR
7440-31-5	Tin	6.6	U		P

Color Before: \_\_\_\_\_ Clarity Before: \_\_\_\_\_ Texture: \_\_\_\_\_

Color After: \_\_\_\_\_ Clarity After: \_\_\_\_\_ Artifacts: \_\_\_\_\_

Comments:

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